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# NAVAL POSTGRADUATE SCHOOL
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# THESIS

Composite Materials at
High Temperatures

by

CHARLES RICHARD BESS

September 1988

Thesis Advisor Ramesh Kolar

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An experimental investigation was made to determine the ultimate tensile strength and elastic modulus of IM6-3501-6 graphite epoxy laminated specimens exposed to temperatures from 70°F to 600°F. Specimens were layered (0°), (90°,0°), (90°,0°,90°). The coupons used are candidate materials for advanced tactical missile airframe design. A Material Testing System (MTS) testing machine with 100,000 lb. maximum load capacity was used to apply the programmed uniaxial tensile loading. A Research Incorporated quad elliptical heating chamber, together with temperature and power controller, were used for maintaining the elevated temperatures. A Measurements Group data acquisition system 4000 was used for data acquisition and data reduction.

The ultimate strength of the 12 plied composite specimen was determined at two temperatures, 70°F and 138°F. The failure of the grips precluded the determination of the modulus at higher temperatures. The overall modulus was obtained for the same type of specimen at several temperatures up to 400°F.


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# Composite Materials at High Temperatures

by

Charles Richard Bess
Lieutenant Commander, United States Navy
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Submitted in partial fulfillment of the
requirements for the degree of

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# ABSTRACT


An experimental investigation was made to determine the ultimate tensile strength and elastic modulus of IM6-3501-6 graphite epoxy laminated specimens exposed to temperatures from 70°F to 600°F. Specimens were layered (0°), (90°,0°), (90°,0°,90°). The coupons used are candidate materials for advanced tactical missile airframe design. A Material Testing System (MTS) testing machine with 100,000 lb. maximum load capacity was used to apply the programmed uniaxial tensile loading. A Research Incorporated quad elliptical heating chamber, together with temperature and power controller, were used for maintaining the elevated temperatures. A Measurements Group data acquisition system 4000 was used for data acquisition and data reduction.

The ultimate strength of the 12 plied composite specimen was determined at two temperatures, 70°F and 138°F. The failure of the grips precluded the determination of the modulus at higher temperatures. The overall modulus was obtained for the same type of specimen at several temperatures up to 400°F.

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# I. INTRODUCTION

There are many increasing important reasons for studying the behavior of high temperature composite materials. The increasing demand for more efficient and reliable thermal machines causes a continuous need for new high temperature materials. Future missiles will be using composite parts in both engines and airframes. These components will be exposed to high temperature environments. The increasing energy costs and supply problems of Chromium, Cobalt, Tantalum, Tungsten, and other high temperature metals continue to stimulate research for alternate materials. Ceramics have excellent high temperature-strength properties but are generally brittle and lose strength catastrophically as a result of mechanical damage. High temperature composites offer the possibility of solving these needs and problems.

The term composite has come to mean a material made by dispersing particles of one or more materials in another material, which forms a substantially continuous network around them. The properties of the composite may bear little relation to those of the components, even though the components retain their integrity within the composite. [Ref. 1]

The ever increasing range and velocities of missiles subject these composite structures to varying temperatures from ambient air to above 350°F. In order to properly use composite materials at increasing temperatures, their performance at these elevated temperatures needs to be known. The loading of structural members in missile power plants may require that the tensile test temperature be reached in a matter of seconds and the test completed to fracture in considerably less than a minute. Under these conditions the following factors must be carefully taken into account in determining the strength of the material. [Ref. 2]

- Loss of strength at elevated temperatures.
- Creep rate of the material during the test.
- Effects of strain rate on the resultant strength level.
- Effects of charring or other physical changes during the test.
- Residual strength after repeated heating and cooling.

The documentation of the strength and stiffness of epoxy based composites is considerable however, more data is needed for specific combinations of layers and orientation at elevated temperatures.

# II. RECENT RESEARCH

Many recent articles have been published in the area of the behavior of composite materials at elevated temperatures. The availability of large numbers of types of fibers and matrix compounds make an almost endless combination of materials. A recent review of pertinent research, although not exhaustive, is presented in this chapter.

Studies by Poveromo [Ref. 3] showed that extensive testing of state-of-the-art epoxies at Grumman (Environmental Sensitivity Program) and other aerospace companies have established a 260°F upper service limit for moisture conditioned graphite/epoxy. This limit was set due to severe degeneration of its mechanical properties above that temperature.

Meyn and Shahinian [Ref. 4] show that there were no significant effects of temperature in the range of 75°F to 250°F and of strain rate on tensile strengths, tensile elastic modulus or in plane shear modulus of the epoxy-graphite composite.

Greszczuk [Ref. 5] found that for T300/5208 graphite epoxy tensile strengths showed little or no reduction until approximately 600°F. His investigation utilized short time heating and thermal exposure.

Kim [Ref. 6] determined that tensile strength increases up to 360°F but decreases above that temperature. The material chosen was 4501-5 graphite epoxy with an orientation of (0/+45/-45/90).

# III. OBJECTIVES AND SCOPE OF THE TEST PROGRAM

## A. OBJECTIVES

The objective of this investigation was two fold. The first task was to acquire and install a data acquisition system to be used with an existing MTS 810 testing machine. The testing system was to be installed and verified in working order. The second objective was to establish short term high temperature tensile strength of IMG-3501-6, 12 plies graphite epoxy specimens exposed to temperatures from 70°F to 600°F.

## B. SCOPE OF THE PROGRAM

The increasing range and life of modern missiles requires the investigation of mechanical properties at high temperatures. Accordingly, IMG-3501-6 based composite coupons were selected for testing and accumulating an experimental data base to determine if this material can be used in the tactical missile airframes subjected to temperatures of 400-600°F over short duration times.

# IV. DESCRIPTION OF THE SPECIMENS

## A. PHYSICAL DESCRIPTION

The specimen dimensions are shown in Figure 1. A total of seven specimens were provided by Naval Weapons Center, China Lake.

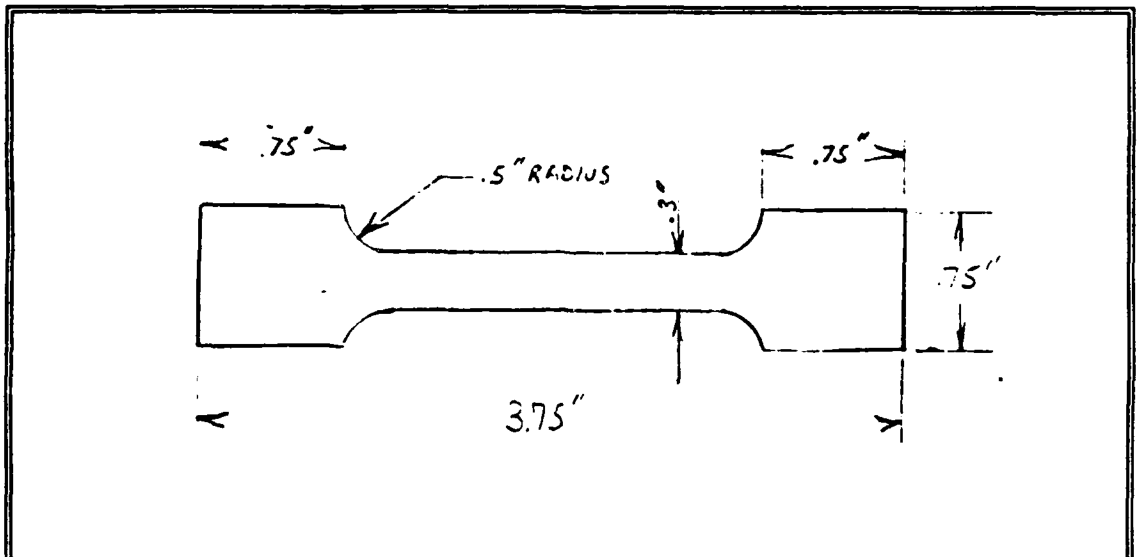



Figure 1. Typical Specimen

## B. SELECTION OF STRAIN GAGES

The discussion of the selection of strain gages, adhesives, application of the gages, and thermal compensation techniques are taken from Reference 8.

The WK series gages manufactured by Measurements Group Incorporated, were selected. They have an integral high endurance lead ribbon with a backing and encapsulation matrix consisting of a high temperature epoxy phenolic resin system reinforced with glass fiber. The temperature range is -452°F to 550°F for continuous use in static measurements. They are useful to +700°F for short term exposure.

## C. SELECTION OF ADHESIVE

Adhesive selection was made based on studies by Tuttle na Brinson. [Ref. 8] All commercially available strain gage adhesives are compatible with epoxy-matrix composite materials. However, a concern arises when adhesives requiring an elevated-temperature cure cycle are used. Among the possible problems in these cases are:

- The glass-transition temperature (Tg) of epoxy-matrix composites is typically in the range of 120-177°C (250-350°F), whereas many of the elevated temperature adhesives are normally cured at or above these temperatures. If the temperature of an unconstrained composite specimen is near (for a long time) or above (for a short time) the Tg during curing, specimen warping often occurs.

- Many of the mechanical properties of polymeric materials, especially the viscoelastic properties, are dramatically affected by previous thermal excursions at or near the Tg. To assure representative and repeatable material response, composite specimens are often subjected to a carefully controlled post cure thermal cycle. This careful thermal conditioning can be destroyed if a gage assembly is subsequently cured at temperatures near the Tg.

- These potential difficulties can often be avoided by using the lowest curing temperatures possible with the adhesive system being used, which usually necessitates long curing times. As an example, appropriate curing times versus curing temperatures published by Micro Measurements for the M-Bond 600 adhesive system are shown in Figure 2. Both a 'recommended' cycle and a 'minimum' cure cycle are indicated.

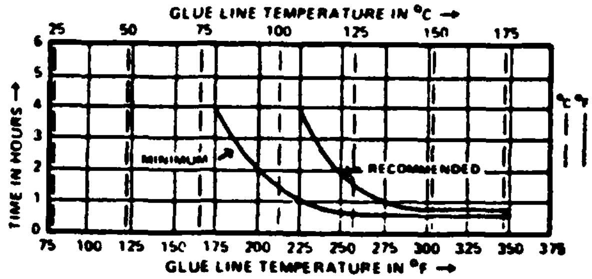



Figure 2. Curing Time [Ref. 8]

## D. ERRORS DUE TO GAGE MISALIGNMENT

A common method of gage alignment when mounting strain gages to conventional structural materials is to first burnish alignment marks on the specimen surface during initial specimen preparation. These alignment marks are usually applied using a relatively blunt-tipped instrument, such as 4-H drafting pencil or a ballpoint ink pen. The strain gage is then aligned using these marks, often with the aid of a short length of transparent tape. While this procedure is quite satisfactory for general purpose strain gage applications, gage alignment cannot be guaranteed to tolerances better than about +/- one to two degrees from the intended gage direction. Under most conditions, gage misalignment of one to two degrees produce negligible measurement errors when mounted to an isotropic material. For a single strain gage mounted to an isotropic material and subjected to a uniform biaxial strain field, the magnitude of the error due to misalignment depends upon three factors (ignoring transverse sensitivity effects):

- The ratio of the algebraic maximum to the algebraic minimum principal strains.
- The angle $\phi$ between the maximum principal strain axis and the intended axis of strain measurement.
- The angular mounting error, $\beta$, between the gage axis after bonding and the intended axis of strain measurement.

These three general conclusions for isotropic materials may be extended to composite materials. However, as in the case of transverse sensitivity effects, the orthotropic nature of composites serve to produce results quite different from those for isotropic material. As an illustration, mount a gage along the major axis of the specimen, but the gage is misaligned some small amount $\beta_0$. For an isotropic specimen, the principal strain directions coincide with the principal stress directions, and hence the gage is aligned very closely with the principal strain directions, since $\beta_0$ is small. The error due to misalignment is therefore small. Now consider a uniaxial tensile specimen of an off-axis composite material whose fibers are at an angle  away from the major axis of the specimen. Again let an axial gage be misaligned a small angle $\beta_0$. Since the off-axis composite specimen is orthotropic, the principal strain directions do not in general coincide with the principal stress directions. Hence, the angle $\phi$ between the maximum principal strain axis and the intended axis of strain measurement is much larger in the orthotropic case than in the isotropic case. The same small misalignment error therefore produces a much larger error in strain measurement for the composite case than for the isotropic case.

As previously mentioned, the use of burnish marks to aid in gage alignment is not possible with epoxy-matrix composites, and hence gages are often mounted to tensile specimens 'by eye.' The cloth-like pattern left by the scrim cloth on many composites can also be misleading, as one is tempted to align the gage using this pattern as a guide, yet this pattern does not necessarily reflect the true fiber direction. These considerations indicate that unless further precautions are taken, gage alignments on composites cannot be held to tolerances better than about +/- two to four degrees from the intended gage direction.

The results for the axial gage are given in Figures 3 and 4. In Figure 3 the numerical error in $\mu$in/in between the actual axial strain $\epsilon_x$ and the measured axial strain $\epsilon'_x$ is presented as a function of fiber angle $\Theta$ , for misalignment errors of -4, -2, +2 and +4 degrees. In Figure 4 this error is expressed as a percentage of the actual axial strain. Note that at fiber angles of 0 and 90 degrees the error due to misalignment is very small, which would be expected, since at these fiber angles the principal strain directions coincide with the principal stress directions. The percentage error due to misalignment is appreciable for fiber angles ranging from about three to 40 degrees, and the error is a maximum for a fiber angle of about eight degrees.

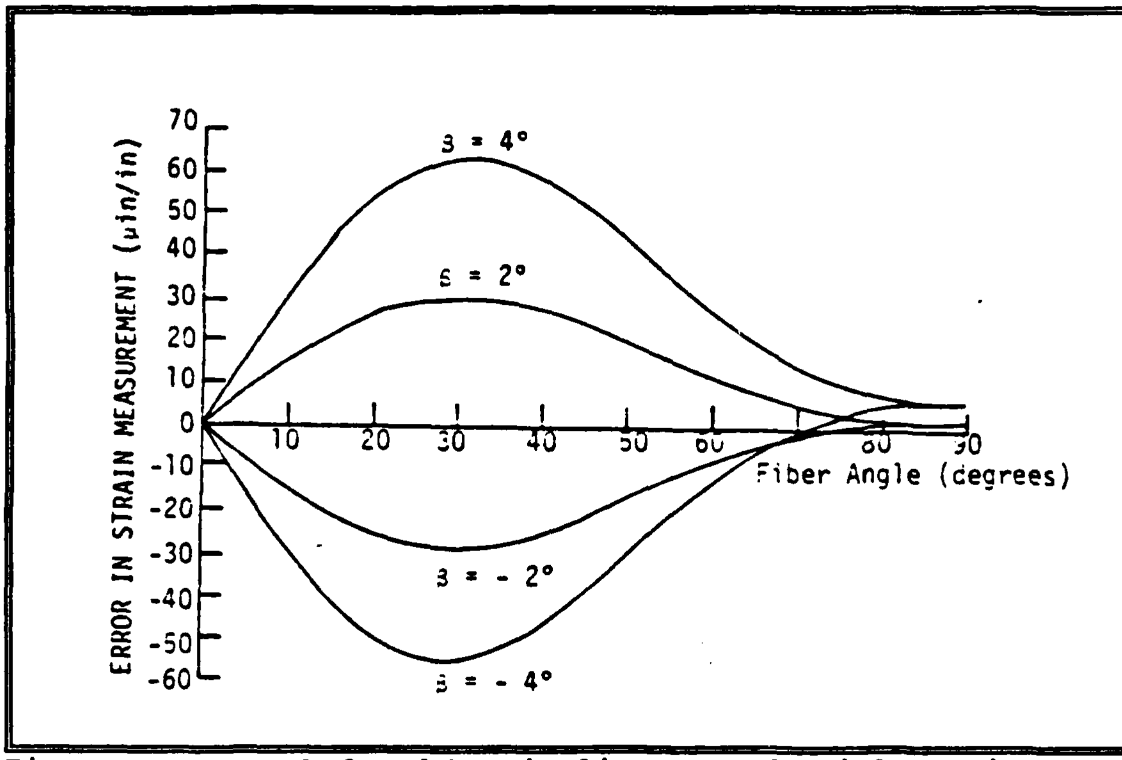



**Figure 3. Error Induced by Misalignment of Axial Strain Gage; Graphite/Epoxy [Ref. 7]**

## E. THERMAL COMPENSATION TECHNIQUES

Of all the potential sources of error associated with the use of strain gages, the most commonly encountered and potentially most serious are those errors due to thermal effects. A change in temperature can affect strain measurement in two ways. The first is that the sensitivity of the gage to strain changes, i.e., the gage factor, changes

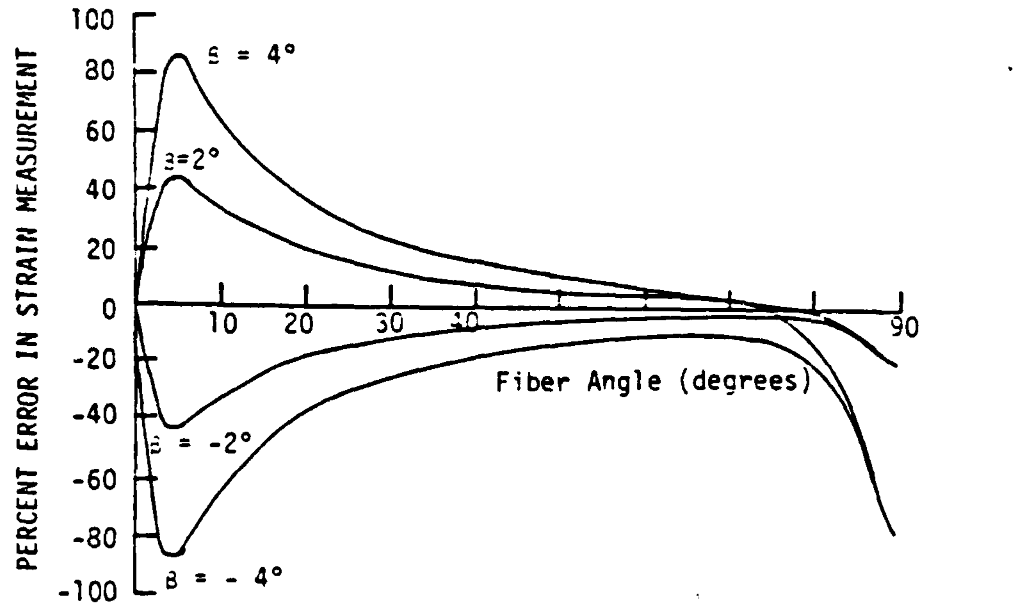



**Figure 4. Percent Error Induced by Misalignment of Axial Strain Gage; Graphite/Epoxy [Ref. 7]**

with changing temperature. Often, the gage factor decreases with increasing temperature, although the opposite can also be true, depending on the particular gage alloy being used. The percentage change in gage factor with temperature is quite small, and is normally on the order of 0.5 per cent per 55°C (100°F). Since this change is so small, gage factor changes with temperature are often neglected.

The second effect due to temperature, and by far the most serious effect, is commonly referred to as 'apparent strain' due to temperature. Apparent strain is the result of several different factors, principally:

- A change with temperature of the electrical resistance of the strain gage.

- A mismatch between the thermal expansion coefficients of the strain gage and the test specimen.
- a change with temperature of the electrical resistance of the strain gage lead wires.

Apparent strain effects can be very large, and if not properly accounted for can completely obliterate the gage response to mechanical loading.

When static or combined static/dynamic strain measurements are made, thermal compensation is almost always required. In modern strain gage practice, two methods of temperature compensation are most often used; self-temperature-compensation (S-T-C) used in conjunction with a three-leadwire system, or temperature compensation using a 'compensating' or 'dummy' strain gage. It should be noted that, at least in theory, thermal compensation is only required when the specimen temperature varies during the course of a strain measurement. However, the level of thermal stability required in order to avoid thermal effects is very difficult to obtain except under closely-controlled laboratory conditions.

## F. THERMAL COMPENSATION USING A DUMMY GAGE

By far the most widely used method of thermal compensation as applied to composite materials is the use of a compensating or dummy gage, in conjunction with the standard Wheatstone bridge circuit. A typical configuration is presented in Figure 5. The 'active' gage is mounted to the composite specimen and is subjected to all the mechanical loads (including thermally-induced mechanical loads) and temperature changes which occur during the course of the test. The 'dummy' gage is mounted to a sample of the composite material, and is placed as physically close to the active gage as possible. Ideally, the dummy gage experiences the same temperature changes as does the active gage, but none of the mechanical loads. Due to the characteristics of the Wheatstone bridge circuit, the apparent strain effects due to the temperature changes cancel, and the output from the active gage is due to mechanical loading only, as desired.

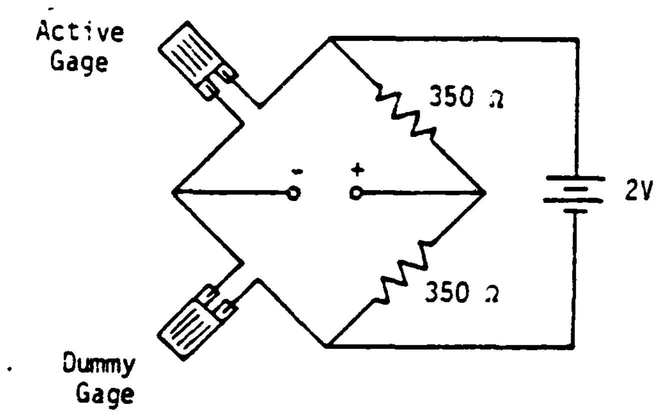



**Figure 5. Dummy Temperature Compensation [Ref. 7]**

## G. CONSTRUCTION OF A TESTABLE SPECIMEN

The first two specimens were instrumented with two strain gages. All others were instrumented with a single strain gage. The gage was principally located along the x axis according to application procedures described in Reference 8. A cure temperature of 300°F was used as recommended. The specimens were cured for one hour at temperature and then allowed to cool very slowly to room temperature over approximately three hours. The strain gage leads were soldered to attachment tabs. Wires to the data acquisition equipment were then soldered next to the gage leads and on top of the attachment tabs (Figure 6).

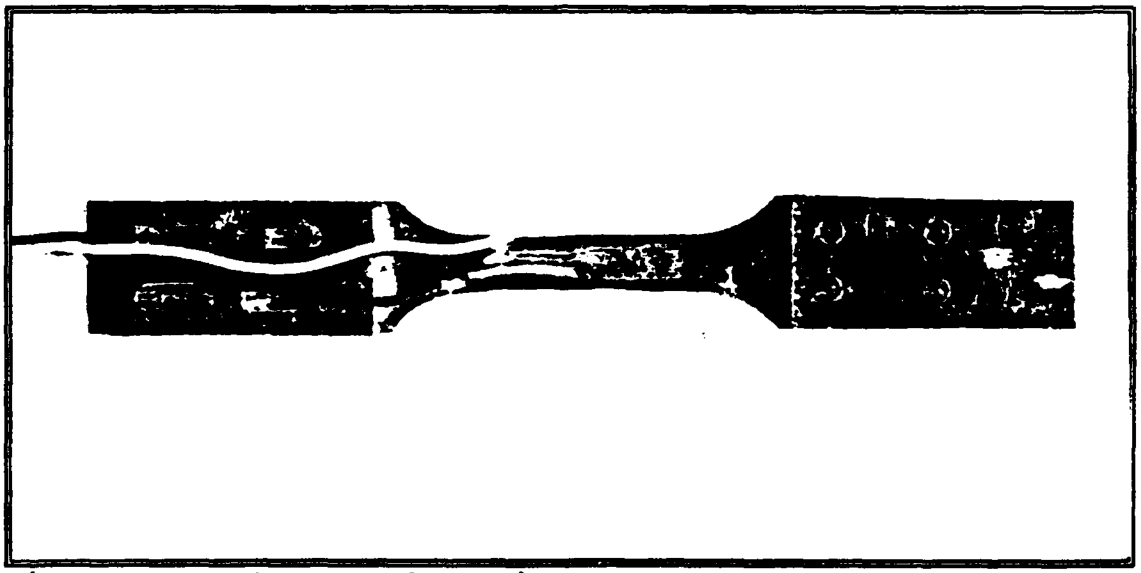


**Figure 6. Instrumented Specimen**

In order to have a good grip on the specimen, aluminum tabs were bonded to the specimen using epoxy cement. The tabs were designed to increase the surface area of the specimen to facilitate gripping (Figure 7). The tabs on the specimens provided were .75 inches x .75 inches. This small area made gripping of the specimen very difficult. To enhance gripping, two aluminum tabs were bonded on each end of the specimen thus increasing the effective area to be gripped. Two different types of epoxies were used, Conley weld and PC.7 multipurpose epoxy paste. The epoxy was mixed according to manufacturer's specifications and applied to all surfaces. The tabs and spacers were then aligned and the excess epoxy was "squeezed" out. A hand clamp was applied and the specimen was allowed to cure overnight.

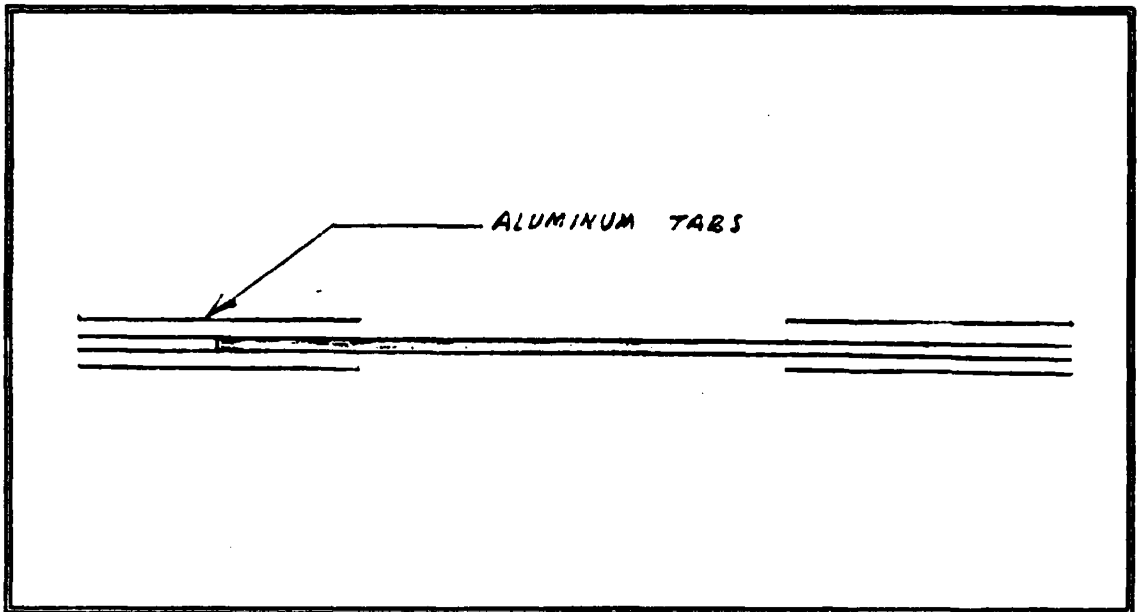



**Figure 7. Specimen with Aluminum Tabs**

# V. EXPERIMENTAL PROCEDURES

## A. TESTING EQUIPMENT

A Materials Test System (MTS) series 810 testing machine with 100,000 pound maximum load capacity was used to apply uniaxial loads. Proper gripping of the specimen is critical to ensure that a uniaxial load is applied. The grips must also be able to hold the specimen throughout its entire test. The grip system used to pull the specimens is described in Appendix A.

A Research Incorporated quad elliptical heating chamber together with temperature controller and power supply were used for maintaining the elevated temperatures. The temperature was controlled by a feedback circuit consisting of a thermocouple constructed from 24 gauge copper constantan wire. The heating chamber is physically two inches in diameter and ten inches long. When the oven was operating, two pieces of aluminum described in Appendix B were placed over the top to prevent heat loss due to air circulation.

The data acquisition system consisted of a Measurements Group, Incorporated System 4000, model 4215 Executive unit. The System 4000 is composed of a central processing unit, four disc drives, keyboard, monochrome monitor, color graphics monitor, dot matrix graphics printer, powerline conditioner, and associated software (Figure 8).

Figure 8. Equipment Set Up

## B. EXPERIMENTAL PROCEDURES

Instrumented specimens were mounted in the grips. Two thermocouples were installed next to the specimen. One thermocouple was for data acquisition and the other was to provide feedback to the oven controller. The dummy gage was placed in the oven and positioned as close as possible to the actual specimen. The strain gages and the load cell were assigned channels on the strain gage scanners. The thermocouples were assigned to the temperature scanners. After the specimen and thermocouples were mounted the data acquisition system was initialized. All channels were verified functional, zeroized, and the data acquisition system was started. Data was taken in intervals of 0.5 seconds or 1.0 seconds. The oven was energized and allowed to reach a steady temperature in approximately 30 to 60 seconds. Due to the limited number of specimens available the following temperatures were chosen for investigation: 70°F, 150°F, 300°F, 400°F, 500°F, and 600°F. Upon reaching the desired temperature, the programmed loading sequence was initiated on the MTS machine. The MTS machine was configured as follows:

- 410 Functional Generator
  Rate 1, set at 60 seconds to give a 30 second rise time. Rate 2, set at 1000 seconds to hold specimen at breakpoint and allow the MTS machine to be shut down. Breakpoint Normal set at 50 percent. RAMP with HOLD AT BREAKPOINT were selected.

- 442 Controller
  LOAD selected with VARIABLE EXCITATION set at 10 percent. SPAN 1 set at 40 percent.

These settings yield a constant ramp load of 2000 lbs, with a rise time of 30 seconds. At the on set of failure, the oven and the MTS machine were quickly turned off to prevent any further damage to the specimen and possibly, the oven and/or the stand holding it.

As the experiment is in progress, the executive unit continuously interrogates all 140 channels and records the respective temperature, strain and load information in parallel. The attached monitor gives a real-time history of the recorded process variables, which enables monitoring the variables in the experiment. The observation of these real-time plots provides very useful information of the state of the experiment, and if any corrective measures are warranted in executing the experiment.

After completion of the experiment, the software REDUCE was used to reduce the raw data into a form compatible with other programs. The program PENPLOTS was used to generated graphics.

# VI. ANALYSIS

## A. ULTIMATE TENSILE STRENGTH

The ultimate tensile strength for the two specimens tested at 70°F and 158°F are 74.6 ksi and 71.4 ksi respectively. The ultimate tensile strength for the other specimens at elevated temperatures was not determined due to a failure of the epoxy bond between the specimen and the aluminum tabs. Appendix C shows the history of the stress and strain for each of the five specimens.

## B. ELASTIC MODULUS

The elastic modulus is plotted in Figure 9. The elastic modulus increased with an increase in temperature up to 400°F. This increase in modulus is in agreement with Reference 7. The modulus was calculated only up to 400°F as explained in Chapter VII. It may be noted that the modulus obtained is for the laminated composite specimen and not for an individual lamina.

## C. CHARRING OF SPECIMENS

The specimens started to char and show signs of delamination at 500°F. At 600°F there was visible smoke from

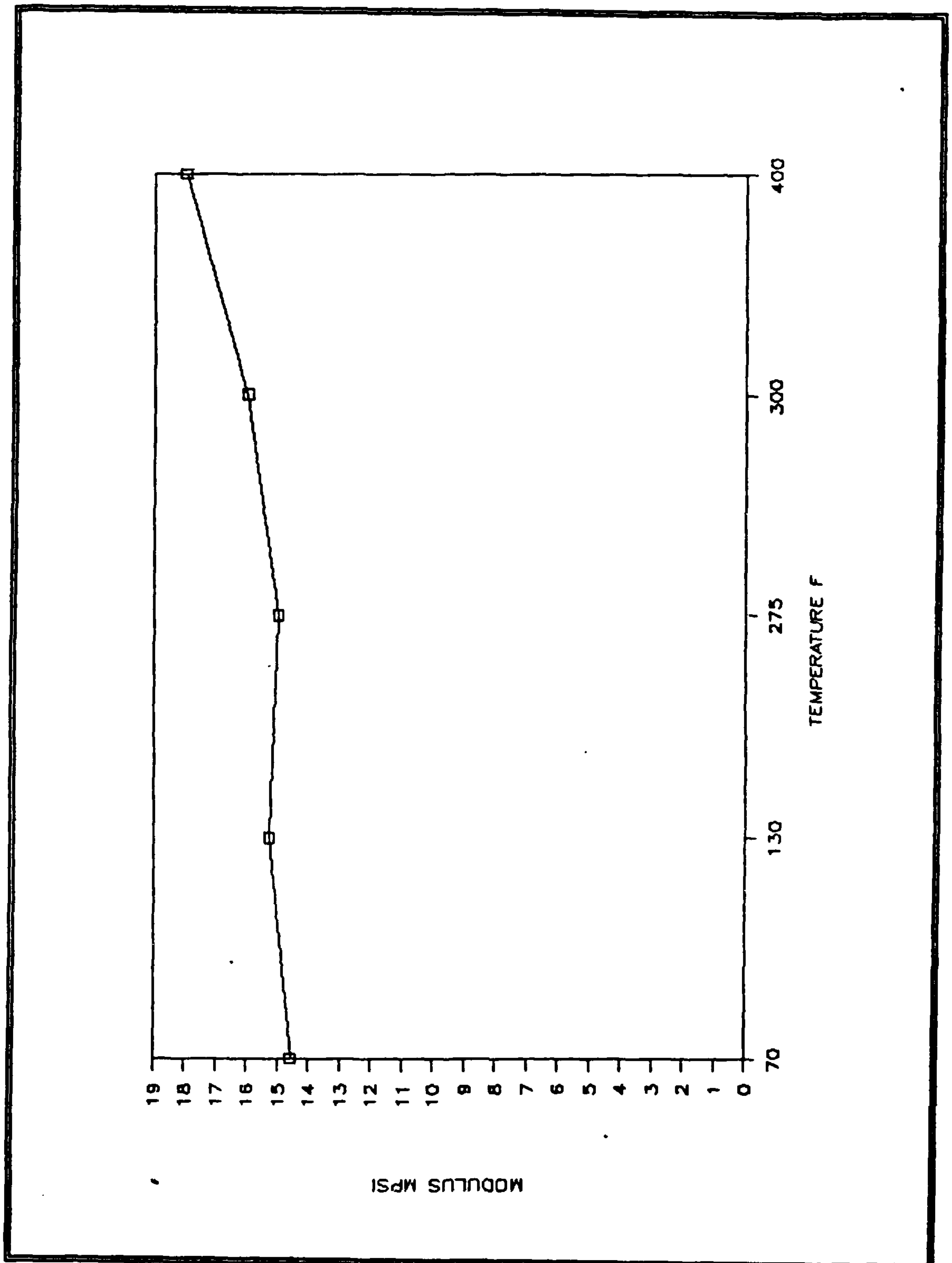



**Figure 9. Modulus Versus Temperature**

the oven. Power to the oven was quickly secured when visible smoke was observed.

# VII. CONCLUSIONS AND RECOMMENDATIONS

## A. CONCLUSIONS

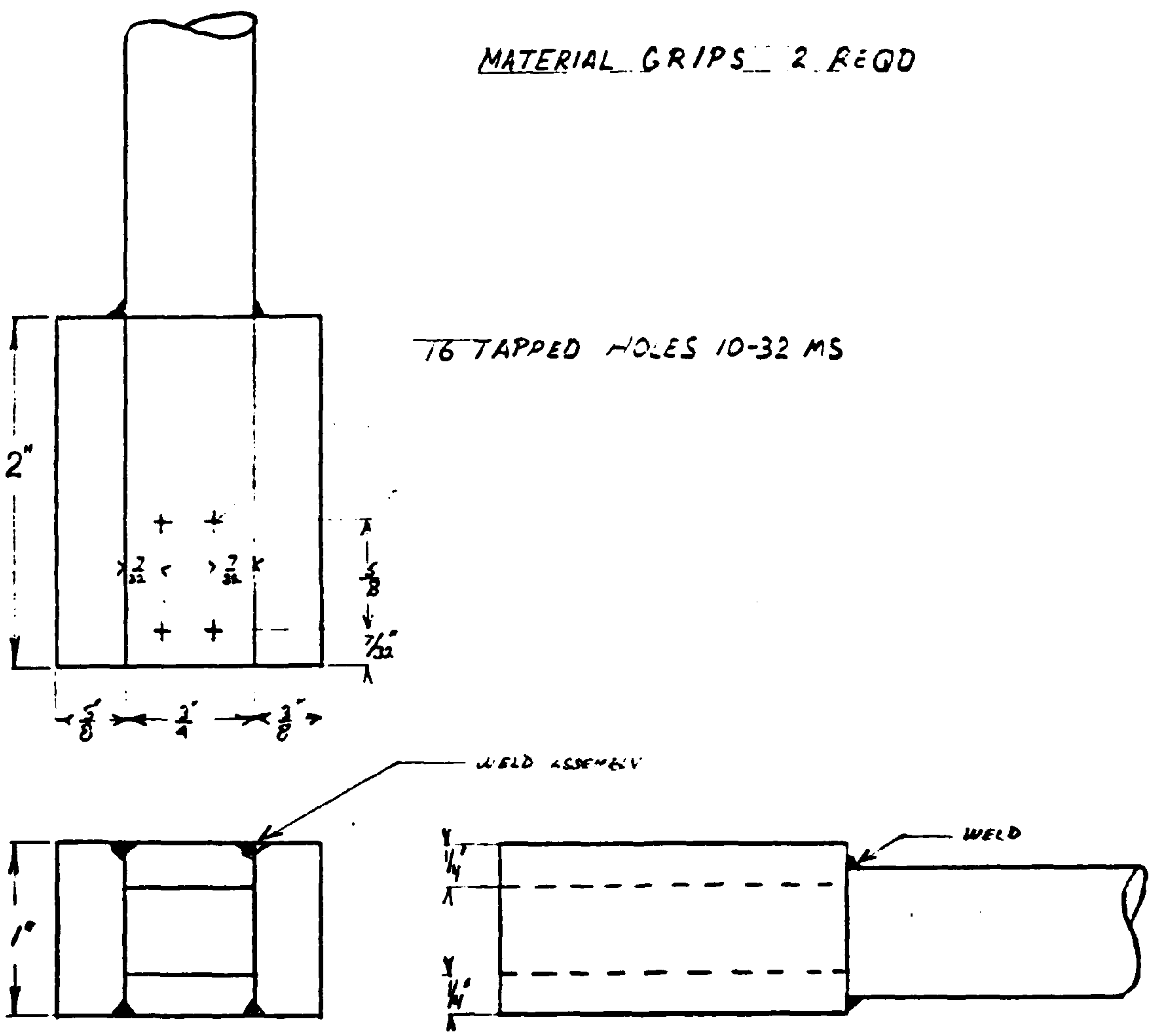
The experimental results agree with the open literature for composites at temperatures up to 300°F. This verifies that the data acquisition system, MTS machine, and oven are all properly integrated. This satisfies the initial objective and prepares the way for more experimentation.

The failure of the grip system is due to the tabs on the specimens being too small. The specimens provided had a gripping surface of only .75 x .75 inches. This small area was not large enough to be held by the mechanical grips as described in Chapter IV, however, the bond between the specimen and the aluminum tabs failed prior to reaching ultimate tensile strength. It is possible that another type of bonding can be utilized to build a larger tab, however, the simplest solution is to manufacture the specimens with a tab of at least two inches long.

## B. RECOMMENDATIONS

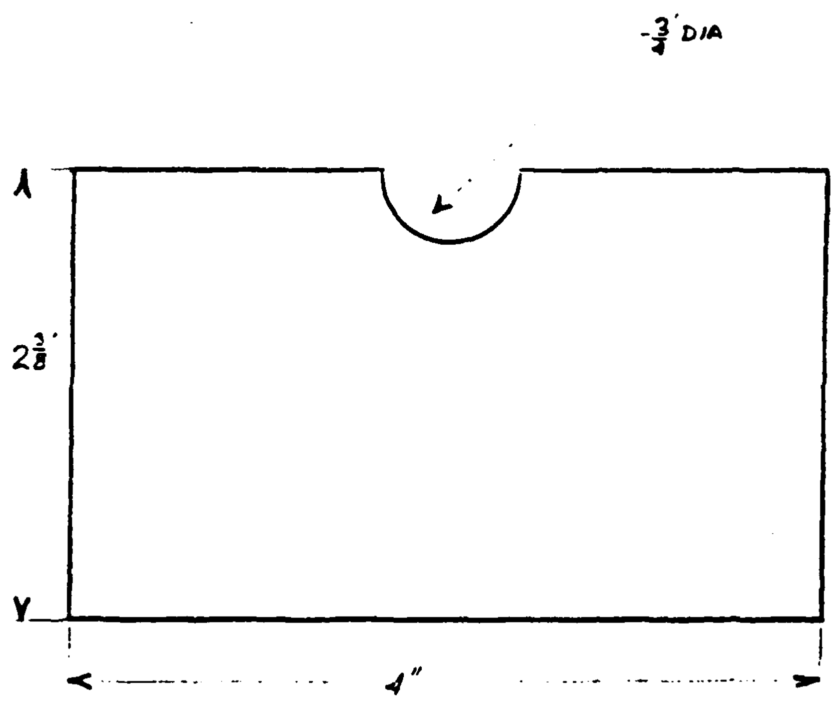
Continued investigation into the effects of high temperature on graphite epoxy laminated specimens is warranted. The equipment set up as described in Chapter V is capable of quickly determining the mechanical properties of specimens at high temperatures at varying load or strain rates. By programming the temperature controller, the effects of the rate of application of temperature may be studied. Also, instead of applying the load, prescribed strains may be applied by appropriate programming of the MTS machine.

# APPENDIX A

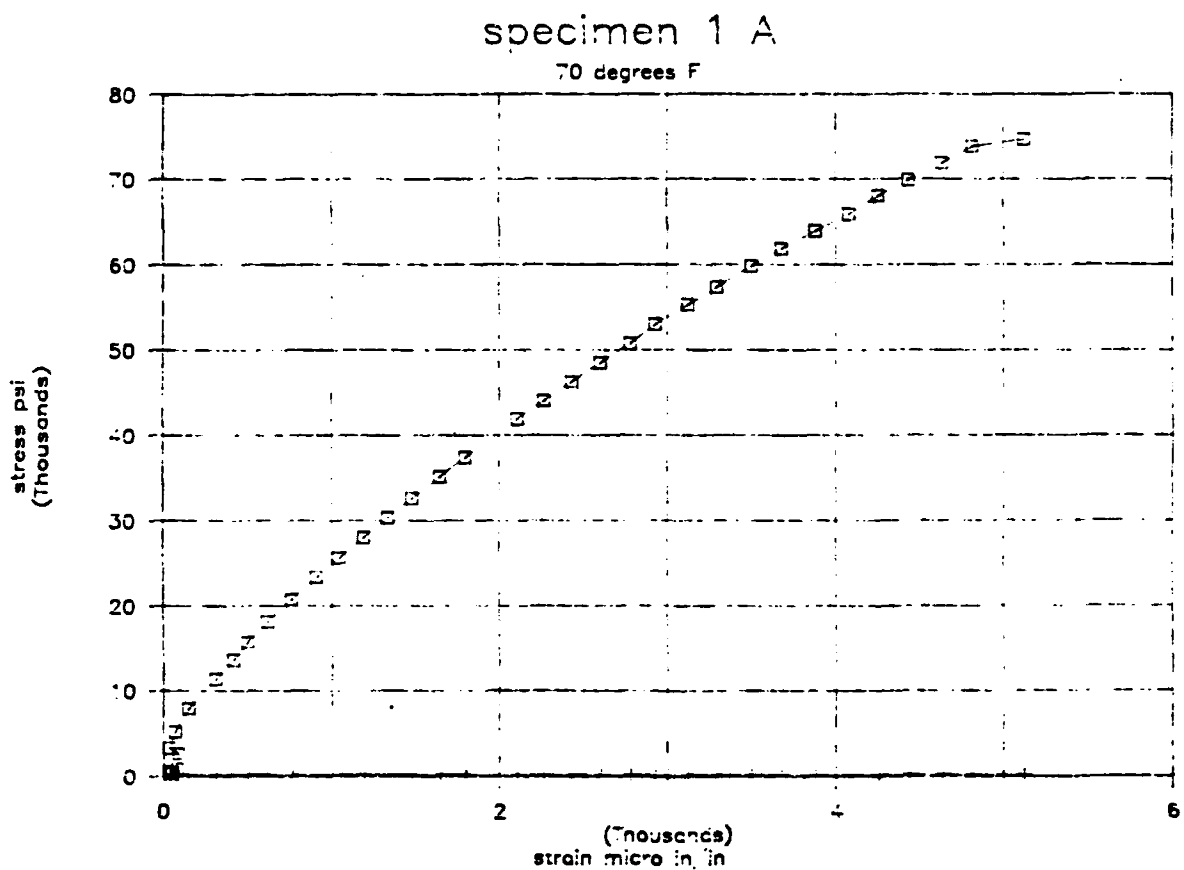

MATERIAL GRIPS 2 REQD
16 TAPPED HOLES 10-32 MS
2"
1"
WELD ASSEMBLY
WELD

# APPENDIX B

OVEN COVER PLATE 2 REQD



# APPENDIX C



specimen 1 A
70 degrees F
stress psi
(Thousands)
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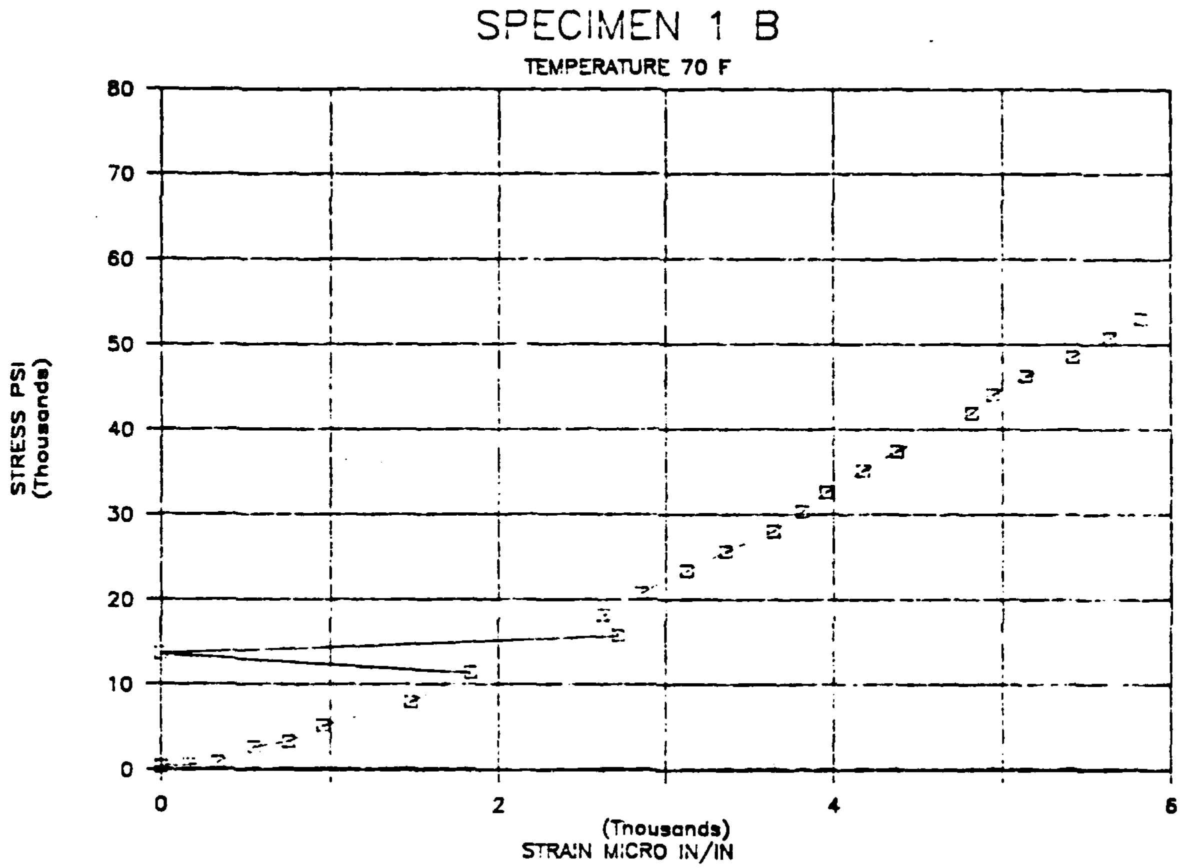
70
60
50
40
30
20
10
0
0
2
4
6
(Thousands)
strain micro in/in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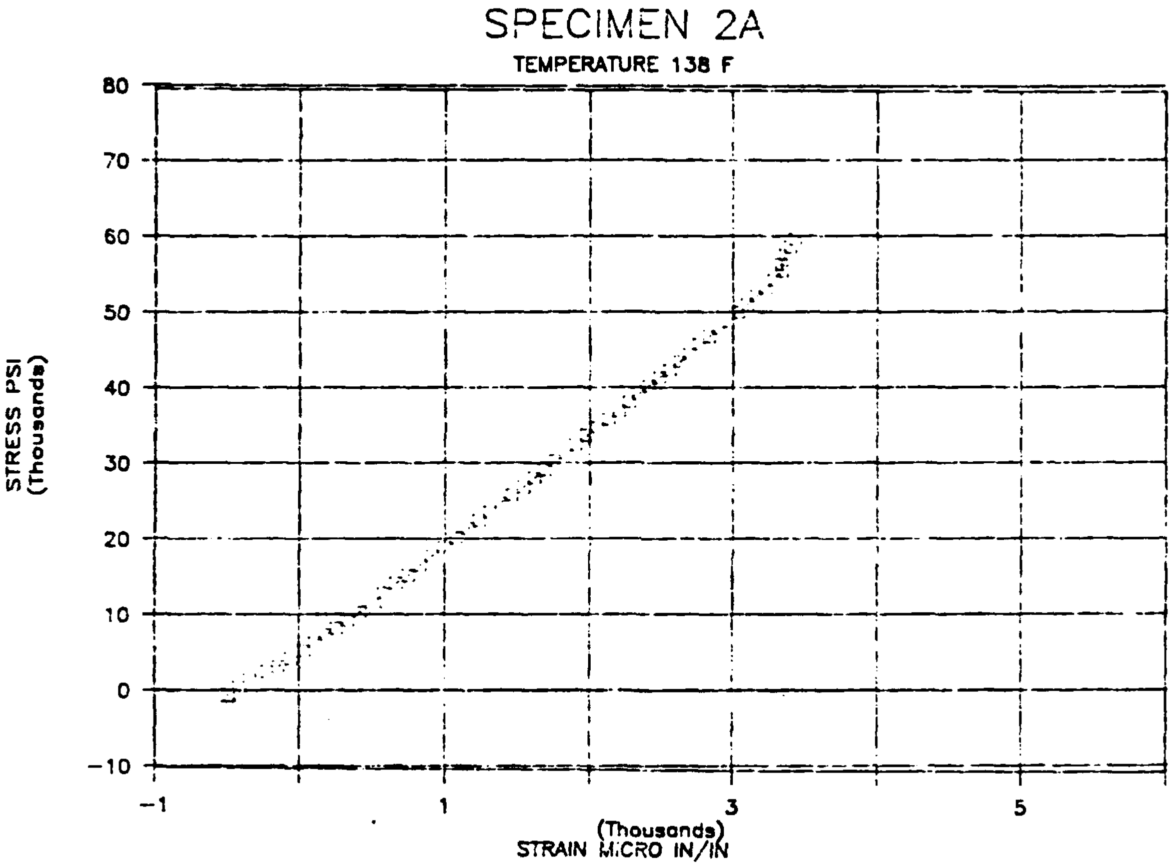
STRAIN MICRO IN/IN



SPECIMEN 2A
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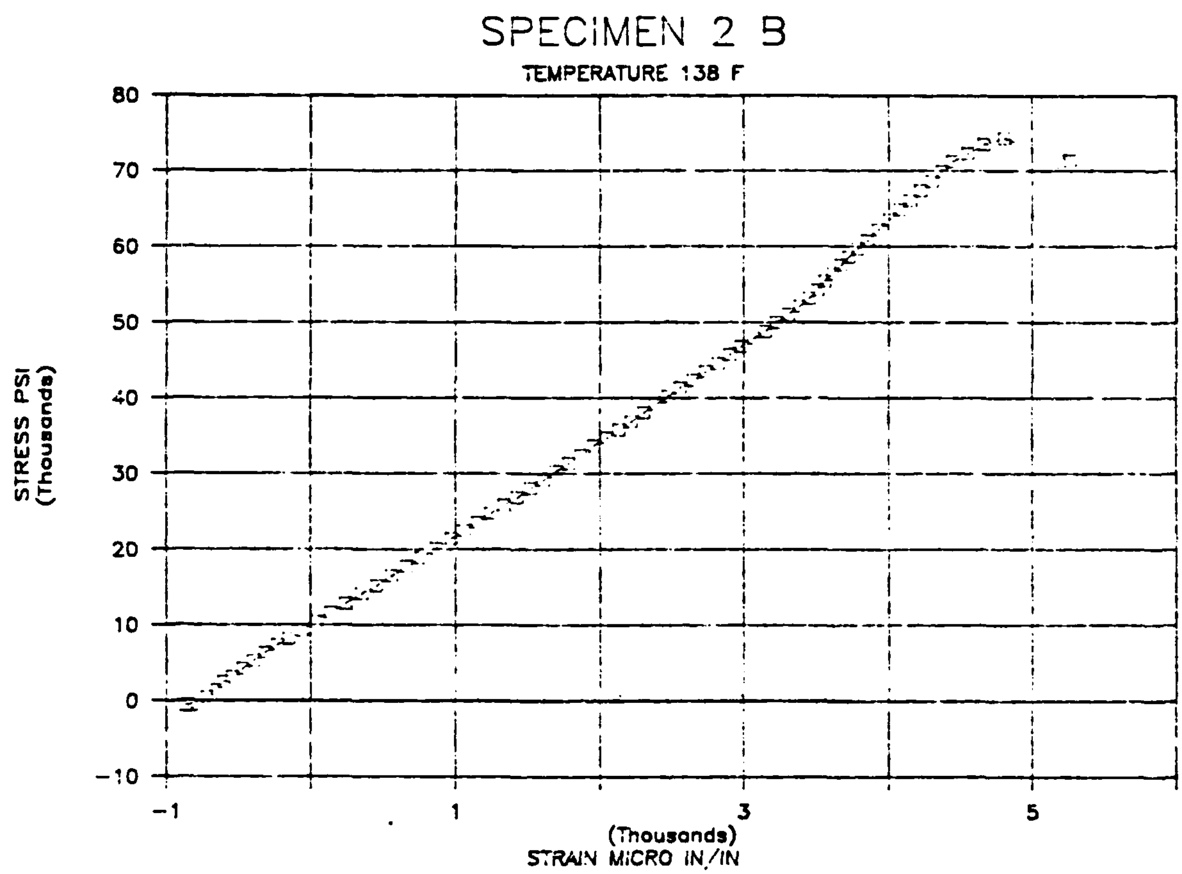
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STRESS PSI
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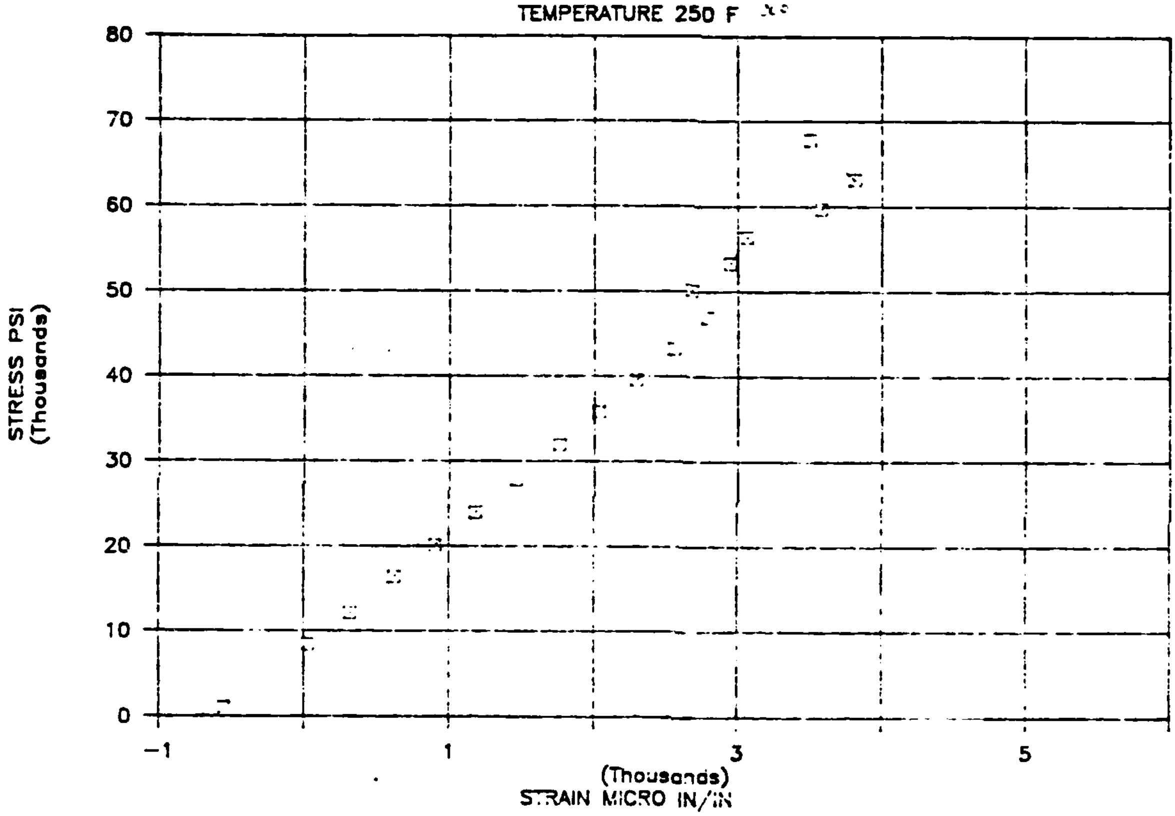
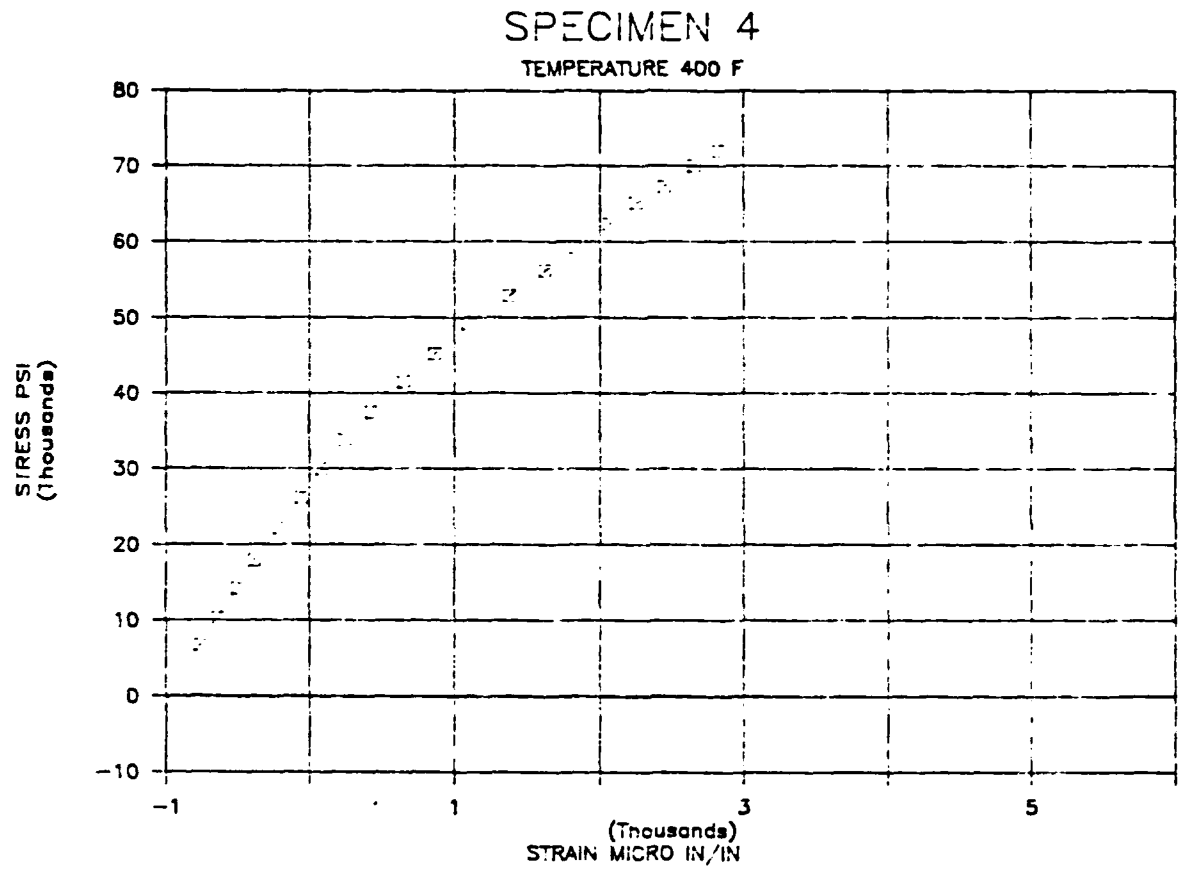
SPECIMEN 3

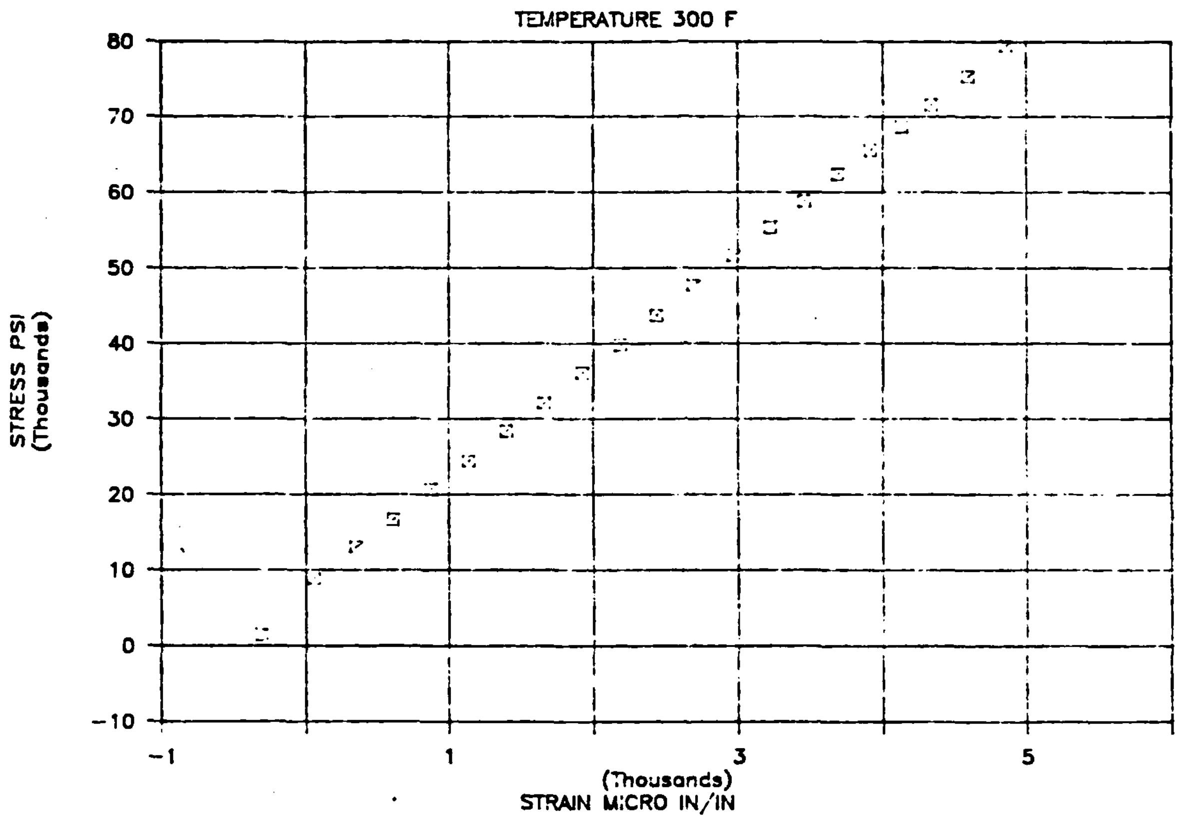
TEMPERATURE 250 F





SPECIMEN 4
TEMPERATURE 400 F
80
70
60
50
40
30
20
10
0
−10
STRESS PSI
(Thousands)
−1
1
3
5
(Thousands)
STRAIN MICRO IN/IN

# SPECIMEN 5



TEMPERATURE 300 F
80
70
60
50
40
30
20
10
0
-10
STRESS PSI
(Thousands)
-1
1
3
5
(Thousands)
STRAIN MICRO IN/IN

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